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54 **Process for the purification of oils.**

57 A process for the purification of oils such as perfluoropolyether oils, silicone oils or oils based on hydrocarbons, which are contaminated with different impurities, in particular, with dispersed solid matter which can be filtered only with difficulty with conventional filters, which process comprises filtration carried out with a filter of the tangential-flow type, the pores of the filter element having a diameter of less than 0.4µm.

1 PROCESS FOR THE PURIFICATION OF OILS

The present invention relates to a process for the purification of oils contaminated with different impurities
5 and, in particular, with dispersed solid matter which can be filtered only with difficulty with conventional filters. The process is useful, for instance, for regenerating oils used as fluids in vacuum pumps and in apparatuses of various kinds wherein, during use, the oil is progressively polluted by foreign substances that may be present in the oil either in solution or in the form of finely-dispersed solid matter. In many cases the dispersed solid substances are either in the form of gels or gums or mucilages which may form a film of practically impermeable
10 matter that drastically reduces the filter efficiency of the filters of the conventional type.

It is known from GB-A-1.561 696 that it is very difficult or almost impossible to eliminate fine particles of
20 foreign materials dispersed in non-conductive liquids, such as, for example, oils, by using a film. The British patent discloses an apparatus for the electrostatic purification of oils by using various collecting members for the impurities, inserted between anode and cathode plates, parallel to the electric field created in the apparatus.
25 The value of the applied voltage is determined by the size of the particles and increases as the particles become smaller. This method is, however, not practical when a large amount of oil has to be purified and, furthermore,
30 it is also very expensive.

Thus, it is an object of the present invention to provide a very simple and efficient process for the purification of oils, which does not require complicated and expensive
35 apparatuses.

1 The process and apparatus according to the present inven-
tion are particularly suitable for the treatment of per-
fluoropolyether oils which are, for instance, used in
vacuum pumps, for thermal tests of electronic apparatuses
5 and in plasma-etching processes employing gaseous agents
such as SiCl_4 , CF_4 , Cl_2 or O_2 .

Perfluoropolyethers employed for these purposes are, for
instance, Fomblin[®] (registered trademark of Montedison),
10 Krytox[®] (Du Pont) and similar products known on the
market by the name Tyreno[®] (Klüber) and Barierta[®], as well
as the perfluoropolyethers produced according to EP-A-
148482.

15 Several methods are known for the purification of perfluoro-
polyethers contaminated with liquid impurities such as
hydrocarbons (mineral oils). Such methods are, usually,
based on extraction by means of solvents or on chemical
treatment for removing traces of water (US-A-4 178 465).
20 These methods, however, do not solve the problem concerning
the removal of solid impurities, in particular, those in
the form of a gel.

The process according to the invention is also suitable
25 for the treatment of other kinds of lubricating oils of
different chemical nature such as silicone oils and those
based on hydrocarbons.

The basic operations involved in the process according
30 to the invention are:

- An optional degassing for removing volatile products,
which is preferably carried out by blowing an inert
gas such as nitrogen into the oil which is kept at room
35 temperature or at a temperature above room temperature.

1 - Filtration for removing the solids in suspension, which
is carried out by using a filter of the tangential-
flow type, wherein the liquid to be filtered is passed
onto the filter surface at high speed, thus avoiding
5 the formation of a deposit of solid material on the
surface itself. The diameter of the pores of the
filter surface has to be very small so as to achieve
a complete separation of the smallest solid particles
as well. Diameters of less than 0.4 μm and, preferably,
10 in the order of about 0,2 μm , are required.

A suitable type of filter for carrying out the filtration
is that produced by Membrana Inc., U.S.A., and marketed
under the names DYNA-SEP[®] and PHARMA-SEP[®]. The filtering
15 surface of such a filter consists of a cylindrical or tu-
bular element made of porous polypropylene, through which
the liquid to be filtered is passed at high speed
and is kept at a pressure above that outside the tubular
surface. On leaving the tubular filtering surface, the
20 filtrate is collected while the liquid to be filtered is
recycled continuously until the concentration of dispersed
solid substances reaches a high value. The pressure
of the liquid to be filtered obviously depends on the cha-
racteristics of the apparatus which may, at the most, be
25 operated at a pressure of up to 4 bar.

The temperature of the liquid is obviously an important
factor as well. A certain temperature limit cannot be
exceeded. This is determined by the thermal resistance
30 of the material of the filter element. For the filters
described above, this temperature limit is 60°C.

The process according to the invention may also comprise
accessory, additional operations such as a previous decan-
35 tation for a preliminary separation of coarser solids and
of liquid, immiscible impurities, if present, and a pre-

1 ceding filtration on a filter capable of separating the
coarser particles in order to lower the solids content
of the liquid to be conveyed to the tangential-flow
filter.

5

After the filtration in the filter of the tangential-flow
type according to the present invention, the perfluoropoly-
ether or the mineral oil can be used directly as a fluid
in vacuum pumps or it can be subjected to neutralization
10 in order to eliminate acid or basic substances that might
be present. In particular, the neutralization of oils
having acid impurities can be carried out with a car-
tridge charged with activated alumina, activated carbon
or sodium carbonate.

15

The following examples illustrate the purification of a
few types of oil in accordance with the present invention.

EXAMPLE 1

20

Product to be purified: 12 kg Fomblin[®] Y L VAC 25/5 oil
used in pumping assemblies of an apparatus for plasma-
etching with SiCl_4 , CF_4 , Cl_2 , O_2 as gaseous agents.
The product, having a light-brown colour, contained solid
25 substances dispersed in the form of a gel. After decan-
tation over a period of three weeks no separation of dis-
tinct phases was achieved. The product, kept at 50°C ,
was degassed in a heated metal container by blowing
135 l/h of nitrogen into the product for 4 hours.

30

The degassed product was then subjected to a pre-filtration
with a filter having a pore diameter of $15\text{ }\mu\text{m}$, at a
pressure of 1.7 bar.

35

Filtration with a DYNA-PHARMA[®] filter which had a filter
element with a pore diameter of $0.2\text{ }\mu\text{m}$, a filter cartridge

1 of about 51 cm in length and a filtering surface of about
19.5 dm² was started.

Operating conditions: Filtration temperature 50 to 55°C;
5 inlet pressure 2.2 bar; outlet pressure 0.8 to 0.9 bar.

The filtration was carried out until 11.3 kg of oil had
been recovered which corresponded to 94% by weight of the
starting product. The average filtration rate was 11.5 l/h.

10

EXAMPLE 2

Product to be purified: about 20 kg of Fomblin[®] Y L VAC
oil coming from an apparatus for ion implantation and con-
15 taminated, inter alia, with PH₃ and AsH₃.

Decantation: After two weeks a distinct liquid phase ha-
ving a brown colour was formed. The main phase was sepa-
rated and pre-filtration with a filter consisting of
20 cellulose nitrate having a pore diameter of 25 µm was
started. The liquid to be filtered was kept at 60°C.

The pre-filtered liquid was then conveyed to a tangential
filter having a filter cartridge as described in example
25 1. The filtration temperature was 60°C, the inlet pressure
2.2 bar and the outlet pressure 0.8 to 0.9 bar.

An average filtration rate of 7.7 l/h was noted.

30 COMPARATIVE EXAMPLE

The pre-filtered liquid was subjected to filtration on
a vacuum filter of the conventional type with a filtering
membrane made of cellulose nitrate having a pore diameter
35 of 0.8 µm. The filtration rate decreased very quickly
and, after 1 hour, the filter was completely clogged and

1 the amount of collected filtrate was negligible.

EXAMPLE 3

5 The product to be purified consisted of 5 kg of Wacker
AN 30[®] silicone oil used in pumping assemblies of an appa-
ratus for plasma-etching, utilizing CCl_4 , SiCl_4 , O_2 , N_2
as gaseous agents. The product had a light-brown colour
and, although it contained dispersed solid matter in the
10 form of a gel, no separation of distinct phases had been
achieved after three weeks.

The product, kept at 50°C , was degassed in a heated metal
container by blowing 135 l/h of nitrogen into the product
15 for 4 hours.

Then filtration with a PHARMA-SEP[®] filter of about 25 cm
length and a filtering surface of 6.32 dm^2 , having a filter
element with a pore diameter of $0.2\text{ }\mu\text{m}$ was started. Ope-
20 rating conditions: Filtration temperature 50°C , inlet
pressure 1.5 bar, outlet pressure 1 bar, average filtra-
tion rate 3 l/h.

The filtration was repeated with the PHARMA-SEP[®] filter
25 described above. However, the working conditions were
changed as follows: inlet pressure 1.8 bar, outlet
pressure 1.2 bar, filtration temperature 50°C . The ave-
rage filtration rate was found to be about 3.6 l/h.

30 In both test series the filter did not give any indication
of obstruction.

EXAMPLE 4

35 The product to be purified consisted of 5 kg of Edwards
15 mineral oil used in pumping assemblies of an apparatus

1 for ion implantation utilizing PH_3 , AsH_3 and N_2 as gaseous
agents. The product had a dark colour and contained solid
matter dispersed in the form of darkly-coloured impurities
and of gels.

5

The product, kept at 50°C , was degassed in a heated metal
container by blowing 135 l/h of nitrogen into the product
for 4 hours.

10 Then filtration with a PHARMA-SEP[®] filter of the same type
as that used in example 3 was started. Operating condi-
tions: filtration temperature 50°C , inlet pressure 1.6
bar, outlet pressure 1 bar, average filtration rate
1.2 l/h.

15

Filtration was repeated with the same PHARMA-SEP[®] filter
described above, however, the working conditions were
changed as follows: inlet pressure 1.4 bar, outlet
pressure 0.8 bar, filtration temperature 50°C . The ave-
20 rage filtration rate was found to be about 1.1 l/h. In
both test series the filter did not give any indication
of obstruction.

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1 CLAIMS:

1. A process for the purification of oils such as per-
fluoropolyether oils, silicone oils or oils based on
5 hydrocarbons which comprises removing by filtration
impurities, in particular, dispersed solid matter
which can be filtered only with difficulty by conven-
tional filters, characterized in that said filtra-
tion is carried out by means of a filter of the
10 tangential-flow type, wherein the pores of the filter
element have a diameter of less than 0.4 μm .
2. A process according to claim 1, wherein a degassing
treatment by introducing a flow of inert gas and/or
15 a pre-filtration on a conventional filter is carried
out before filtration on the tangential-flow filter.
3. A process according to claim 1 or 2, wherein the
tangential-flow filter is provided with a filter
20 element having a pore diameter of about 0.2 μm .
4. A process according to any one of claims 1 to 3,
wherein the filter element consists of porous poly-
propylene.
- 25 5. A process according to any one of claims 1 to 4,
wherein the oil to be purified is subjected to pre-
vious decantation so as to remove liquid impurities
immiscible with the oil, if present.

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